SMC-TR-03-25

AEROSPACE REPORT NO.
TR-2003(1465)-1

Electrical Resistivity of DC93-500 Silicone Adhesive

10 April 2003

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20031001 225

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This report was submitted by The Aerospace Corporation, El Segundo, CA 90245-4691, under Contract No. F04701-00-C-0009 with the Space and Missile Systems Center, 2430 E. El Segundo Blvd., Los Angeles Air Force Base, CA 90245. It was reviewed and approved for The Aerospace Corporation by B. Jaduszliwer, Principal Director, Electronics and Photonics Laboratory; and S. A. Kaminski, Principal Director, Mission Control Directorate. Robert Bresnick was the project officer for the program.

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Robert Bresnick

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SMC/MC

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. REPORT DATE *(DD-MM-YYYY)* 10-04-2003	**2. REPORT TYPE**	**3. DATES COVERED *(From - To)***
4. TITLE AND SUBTITLE Electrical Resistivity of DC93-500 Silicone Adhesive		**5a. CONTRACT NUMBER** F04701-00-C-0009
		5b. GRANT NUMBER
		5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S) B. A. Morgan		**5d. PROJECT NUMBER**
		5e. TASK NUMBER
		5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) The Aerospace Corporation, Laboratory Operations, El Segundo, CA 90245-4691		**8. PERFORMING ORGANIZATION REPORT NUMBER** TR-2003(1465)-1
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Space and Missile Systems Center, Air Force Space Command, 2450 E. El Segundo Blvd., Los Angeles Air Force Base, CA 90245		**10. SPONSOR/MONITOR'S ACRONYM(S)** SMC
		11. SPONSOR/MONITOR'S REPORT NUMBER(S) SMC-TR-03-25

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

Dow Corning 93-500 clear silicone adhesive is used to attach protective cover glass material to solar cells. If the resistance of the DC93-500 becomes too great as temperatures approach –150°C, then there is the potential for arc discharge, which could damage spacecraft electronics. Therefore, the question of electrical resistance as a function of temperature is important to the design process.

In this report, measurements of resistivity are reported as a function of temperature. A change of slope in the resistivity versus log(temperature) graph at approximately –50°C is reported. This phenomenon is linked to the crystalline phase transition.

15. SUBJECT TERMS

Low-temperature silicone resistivity

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Brent Morgan
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED		9	**19b. TELEPHONE NUMBER *(include area code)*** (310)336-1796

Acknowledgment

Thanks to Tom Giants of The Aerospace Corporation.

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1. Introduction

Spacecraft charging due to the solar wind is a well-known phenomenon. Design precautions must be taken to mitigate damage due to arc discharge, which will occur when electrical potential between two nearby areas on a spacecraft differs by more than the breakdown potential between them. In efforts to mitigate electrostatic discharge damage due to this phenomenon, designers must be able to predict likely potential differences. Knowledge of materials' temperature dependence of resistivity aids in this prediction. Resistivity is generally increasing with decreasing temperature. For most materials, this dependence is known or can be predicted with good certainty.

Dow Corning 93-500 clear silicone adhesive is used to attach protective cover glass material to solar cells. During eclipse, it was postulated that the temperature of the cells and adhesive might reach as low as –150°C. If the resistance of the DC93-500 becomes too great at those temperatures, then there is the potential for arc discharge, which could be damaging to spacecraft electronics. Therefore, the question of electrical resistance as a function of temperature became important to the design process.

The known properties of 93-500 include the glass-transition temperature at about –120°C (~95 on an Arrhenius plot using eV as its energy scale) and a largely ignored crystalline transition temperature at about –50°C (~46 on the 1/kT scale when seen on an Arrhenius plot using eV as its energy scale). The crystalline transition is reported to be dependent upon the rate at which the temperature drops in the vicinity of –50°C; slower rates are thought to allow the onset of a crystalline phase, while faster rates freeze in the native room-temperature amorphous phase.

A government contractor reported measurements of DC93-500 resistivity, taken at 25°C increments starting from 50°C and working downward. Disagreement between the several data sets that were reported was puzzling, and program management wanted to validate the contractor's estimates of the resistivity.

2. Measurement Techniques and Experimental Setup

A single rectangular piece of DC93-500 was used for all measurements with dimensions approximately 11.5 cm by 11.5 cm by 0.24 cm. The sheet was cast at the contractor's facility, using standard methods. The sheet was cleaned at various times with isopropanol.

All resistivity measurements were performed with a circular guarded electrode through the 0.24 cm dimension of the silicone to prevent contributions from surface conduction on one side and a large rectangular plate on the other. This precaution, using a guarded electrode, was perhaps unnecessary since surface conduction seems not to be an issue in this experiment. A pair of data runs by the contractor, differing only in the use of a guard ring, showed little real difference in conductivity. In later data runs at Aerospace, total power supply current was monitored to determine whether leakage current increased with decreasing temperature, a sign of possible increased surface conduction. We found that it did not increase over the duration of the experiment within the resolution of the power supply voltmeter (EG&G/Ortec model 556 with a current resolution of 10 μA). Nonetheless, we thought it prudent to take what precautions we could to guard against surface leakage, and all measurements reported herein were taken with the guard ring in place.

A Keithly electrometer (model 617) was used to integrate charge between top and bottom electrodes for a known length of time. The length of integration was chosen so as to give relatively low noise results—30 s to 1 min near room temperature, to tens of minutes at low temperature. Simple division gives the average current, and the dimensions of the top electrode plus the thickness of the silicone allow one to compute resistivity.

Temperature was monitored on the bottom electrode using a thermocouple probe. Since the heat capacity of the bottom electrode was quite high, it was assumed to accurately represent the temperature of the sample under test. Braided copper cables run from the bottom electrode to a liquid-nitrogen bath beneath. Boil-off from the LN_2 kept vapor from condensing on the samples

Both Dow Corning and the government contractor used an ASTM method to measure resistance. This method also uses a guarded electrode, but the methodology of the ASTM measurement differs from most of the results reported here in one significant way: In the ASTM technique, the bias is removed in between measurements. Using this method, a slightly lower resistance will be measured than with a continuously applied bias due to charge trapping in the silicone. The theory behind this phenomenon holds that the silicone has many sites, both spatially and energetically distributed in the material, that will attract and hold electrons. The initial surge of current into the material is due to filling of the trap sites. The traps will, on average, stay filled only if a constant bias is applied. When bias is suddenly applied or increased, electrons can fill additional trap sites that were previously energetically unfavorable, thereby taking those electrons out of the conduction loop and resulting in an apparent higher net conduction current. When bias is decreased, trap sites at the highest energies lose their electrons, resulting in a net decrease in current. Charge trapping for DC93-500 has a time scale of minutes and can be observed in Figure 1 as the change in current with time at applied constant voltage.

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However, real-life conditions of the silicone adhesive will have it under a slowly varying bias, and therefore all other measurements made at Aerospace are taken under conditions of constant applied bias, with an initial pre-soak. This allows a better estimate of DC resistivity to be made since the constant-bias condition more closely simulates real-world conditions. Typically, the silicone adhesive was soaked at 1000 V for at least 30 min before taking resistivity measurements, often longer.

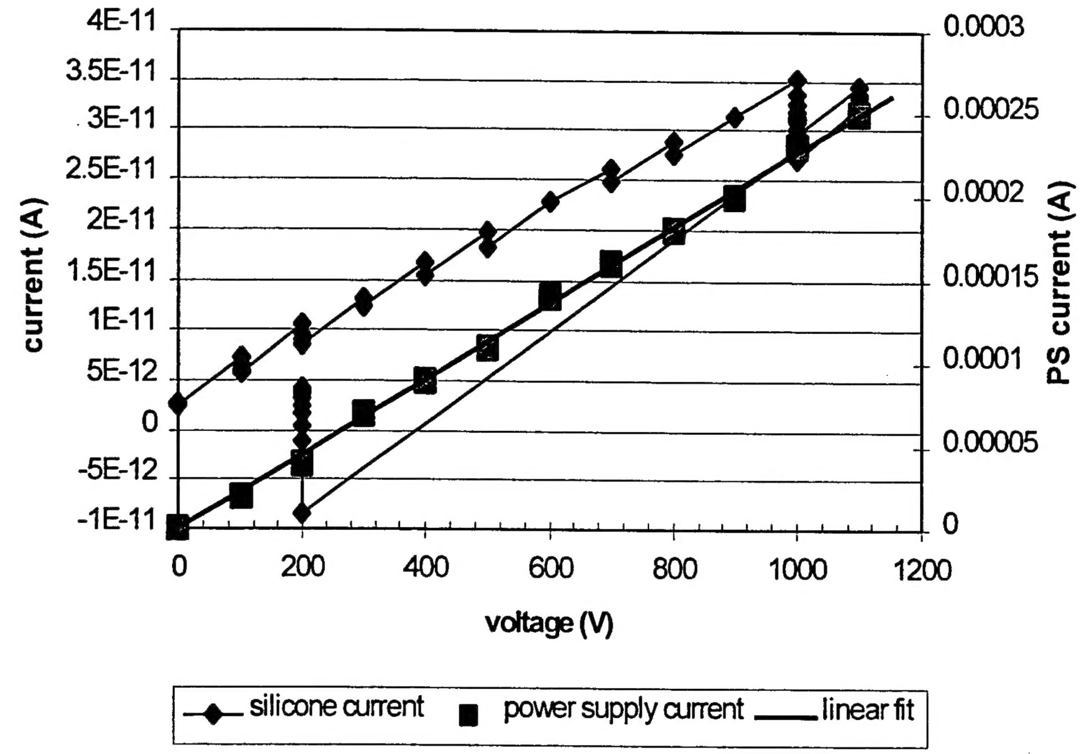

Figure 1. The room-temperature I-V characteristics of DC93-500. This data verifies that the IV relationship is essentially linear at room temperature.

3. Room-Temperature Properties

Figure 1 shows the room temperature current-voltage relationship for the DC93-500 sample. Data in the figure were acquired by ramping the bias from 0 V to 1100V, and then back to 200V, with approximately 1 minute of acquisition time at each data point shown. Overall leakage current (taken from the power supply current meter) is linear with applied bias. The conduction current is also roughly linear with applied bias, but shows some interesting characteristics.

Upon initial step increase of the voltage applied to the sample, the current is higher than at the same bias several minutes later. This effect is due to charge trapping, and is discussed in the previous section. The phenomenon also works in reverse; when bias is decreased from 1100V to 200V, current is initially negative, due to outflow of trapped charge. Neglecting fluctuations due to charge trapping, the current-voltage relationship seems to be quite linear, indicating an absence of non-ohmic conduction mechanisms.

The official DOW measurement of room temperature resistivity is made with the ASTM method, as discussed previously. One can see in Figure 2 that DOW, the government contractor, and Aerospace measurements at room temperature and made with the ASTM method or an ASTM-like method all agree within an acceptable margin (~1e15 Ohm-cm at ~38 on the 1/KT scale). This comparison validates the basic experimental setup.

For the remaining data, it is an assumption that given a sufficiently long soak at fixed bias, one may measure the true DC component of conductivity.

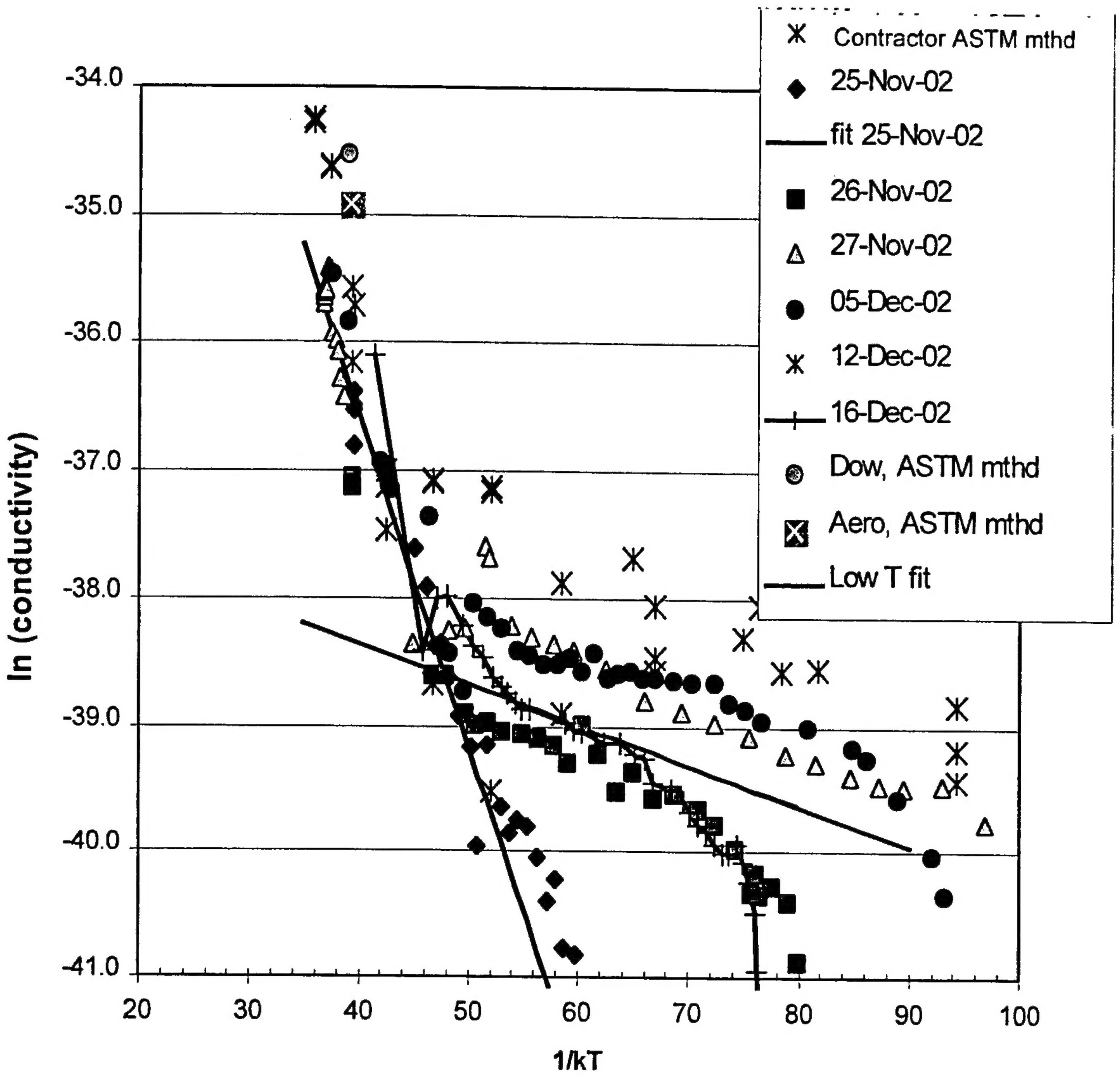

Figure 2. Log conductivity versus kT. The best fit line to 25 Nov 02 data indicates an activation energy of 0.258 eV. The line has an offset of about –26.24. At lower temperatures, the activation energy is about 0.032 eV

4. Temperature-Dependent Properties

Temperature dependence of the resistivity of DC93-500 shows good agreement between the results of the contractor and Aerospace. At temperatures below room temperature, what initially appeared to be great differences in resistivity have been shown to actually be normal variations due to a metastable phase transition in the vicinity of –50°C. Specifically, three of the contractor's four measurements (known as "retest," "guarded," and "unguarded") agree quite well with Aerospace measurements, particularly if one keeps in mind the difference in results that is expected using the ASTM versus constant-bias measurement. (The contractor's data is expected to show a slightly lower resistance due to test methods, all else being equal). A fourth contractor dataset (known as "test" or "earlier test") is also in partial agreement. However, its lower temperature data points are in disagreement with all the other measurements and should be discarded as non-physical since they have a monotonic decrease of resistance below –50°C.

The temperature dependence of the resistance is easily understood. In the range of 50°C to about –50°C, the resistance decreases with activation energy of 0.25 eV. For temperatures around –50°C, resitivity can be widely variant, at times seemingly non-physical: The unwary may even measure a "negative" resistance near that temperature (when accumulated charge is expelled from the silicone during the phase transition, resulting in a negative current). This behavior is assumed to be due to the crystalline phase transition that is known to occur at about –50°C. This phase transition appears to be metastable, thus there is a wide variation in resistivity near this temperature, depending upon crystalline phase and measurement parameters.

In the temperature range of about –50°C to –150°C, the resistance can vary, presumably depending upon the degree to which the material earlier crystallized. If the crystalline transition were incomplete to one degree or another, this sort of variation is to be expected.

Figure 2 is an Arrhenius plot of conductivity. Normalizing the horizontal axis to units of energy (here shown as eV) allows one to directly compute the activation energy of the process as the slope of the graph. Since tradition holds that the slope of the data should be negative, the data is presented as sigma (mho-cm) instead of rho (ohm-cm).

The same data are graphed in Figure 3, but presented as resistivity (ohm-cm) versus temperature.

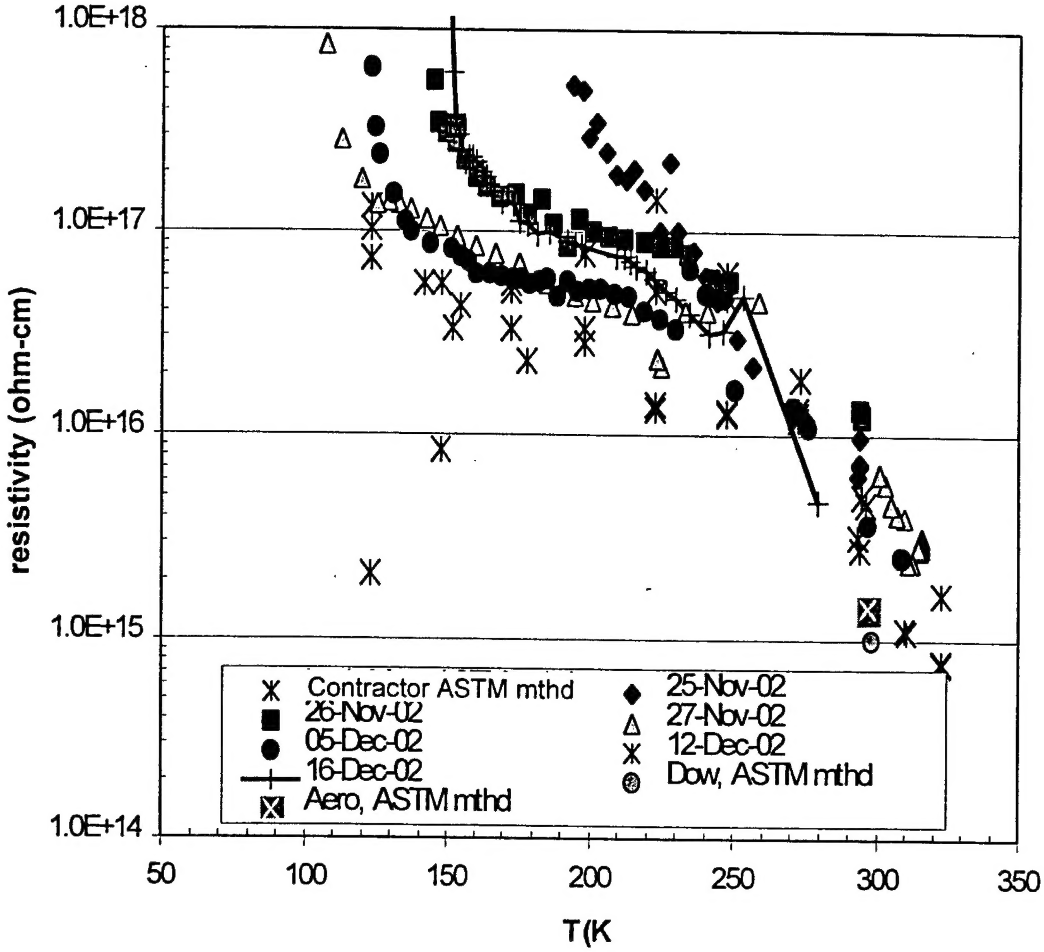

Figure 3. Resistivity vs temperature. This graph presents the data from Figure 2 in a different format. Included for completeness in this graph are two outlying data points (at 125 and 150K) that are believed to be non-physical.

5. Conclusions

DC93-500 silicone resistivity will rise to about 10^{18} Ω-cm at −150°C (~95 on the 1/kT scale). However, depending upon the phase of the material, resistance may be much higher. Based on the work to date, it is unclear what will trigger the phase transition.

With the exception of one of the contractor's datasets, all measurements have been shown to be within an expected range of values. Recognition of this agreement was initially complicated by the change of slope in the resistivity versus temperature graph due to a little-studied metastable crystalline phase transition at −50°C. With the proper background information and appropriately dense data collection, the apparent differences between datasets can be easily explained.

This material and others like it should be studied more extensively. Drs. Gary Stupian and Martin Leung of The Aerospace Corporation have suggested that the silicone itself may have an intrinsic polarization. This might be detected by preserving orientation when making resistivity measurements, something that was neglected in this experiment. There is also the possibility that the past history of the silicone can promote or hinder the crystalline-phase formation.

LABORATORY OPERATIONS

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